

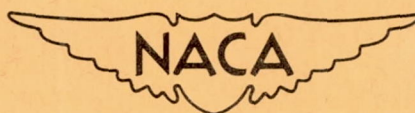
NATIONAL ADVISORY COMMITTEE
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TECHNICAL NOTE 2050

PROPERTIES OF A BORON CARBIDE-IRON CERAMAL

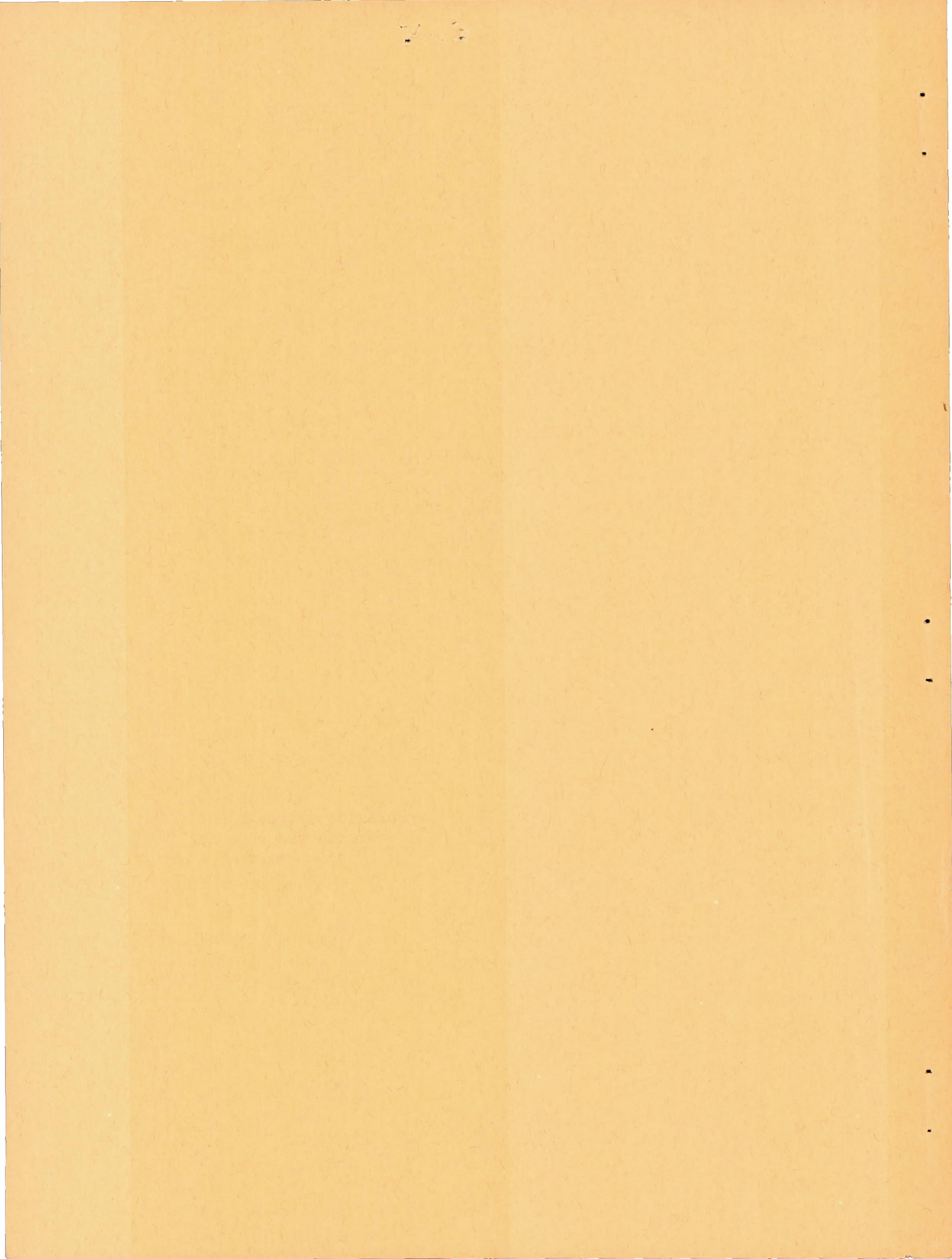
By W. G. Lidman and H. J. Hamjian

Lewis Flight Propulsion Laboratory
Cleveland, Ohio



Washington
March 1950

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PROPERTIES OF A BORON CARBIDE-IRON CERAMAL

By W. G. Lidman and H. J. Hamjian

SUMMARY

An investigation was made to determine the properties of a boron carbide-iron ceramal. These properties were compared with the properties of hot-pressed boron carbide. The properties evaluated were room- and elevated-temperature modulus of rupture, resistance to fracture by thermal shock, density, and oxidation characteristics. A study was also made of the microstructure of the ceramal.

Microscopic examination showed the ceramal to be composed of not less than two phases: boron carbide, and a solution or chemical combination of iron and boron carbide. Ceramals containing materials that form a bonding phase may possess desirable strength properties at temperatures approaching the melting point of the metal.

The hot-pressed boron carbide was superior to the ceramal in modulus of rupture. A high percentage of the room-temperature strength was retained by the ceramal at 2600° F, which was attributed to the high strength of the carbide network, strengthening of the iron by combination with some of the boron carbide, or both. The thermal-shock resistance of boron carbide was improved by the addition of iron. At 1600° F, the resistance to oxidation of the ceramal was better than that of the ceramic; although both materials oxidized rapidly at 2000° F, the ceramic was superior. The poor oxidation resistance of the ceramal indicated that a protective coating would be required for continued operation above 1600° F.

The boron carbide-iron ceramal has a low density (average density 3.24 gm/ml), which gives it an additional advantage when it is compared with metals or other ceramals on the basis of the strength-density ratio.

INTRODUCTION

Designers of flight-propulsion units are currently seeking materials for use in the temperature range from 1800° to 2400° F. Suitable materials must retain their strength in this range long enough to permit reasonable overhaul periods and should be resistant to oxidation and to fracture by thermal shock. Materials with high strength-density ratio are desirable for use in rotating parts. The alloys currently used do not operate successfully above 1800° F and it has become necessary to consider more refractory materials, such as ceramics. It has been found that pure ceramic bodies are unsuitable for this use because they do not possess adequate thermal- and mechanical-shock resistance. Mixtures of ceramic and metal, ceramals, are therefore of interest because the metal additions to the ceramic impart resistance to fracture by thermal and mechanical shock (reference 1).

A previous investigation (reference 2) showed that boron carbide forms a bonding phase with iron, nickel, or cobalt. It was hypothesized that materials which bond in this manner may be used at temperatures above the softening temperature of the metal. An investigation was therefore undertaken at the NACA Lewis laboratory to verify this hypothesis and to determine physical properties of a boron carbide-iron ceramal in the temperature range that is of interest to designers. Iron was selected as the metal addition to boron carbide because it is domestically abundant.

This investigation was limited to a study of a single composition because of the difficulties inherent in devising fabrication techniques for materials of this type. All ceramal specimens used in this investigation were fabricated at the Lewis laboratory in order that the processing variables could be closely controlled. The choice of a composition for investigation is, of necessity, somewhat arbitrary. Previous work on ceramals (reference 3), however, indicates that the metal-rich phase must fill the interstices between the ceramic particles. In order to have sufficient metal present, the amount necessary should be calculated on a volume basis. A titanium carbide-cobalt ceramal, which has about 13-percent metal by volume (20-percent by weight), has good elevated-temperature strength (reference 4) and good resistance to fracture by thermal shock (reference 5). On this basis, a boron carbide-iron ceramal, which contains approximately this same amount of metal by volume, was selected for investigation. The properties evaluated were density, room- and elevated-temperature modulus of rupture, resistance to fracture by thermal shock, and oxidation

resistance. For comparison purposes, pure boron carbide in the form of commercially prepared hot-pressed specimens was also evaluated.

APPARATUS AND PROCEDURE

The results of extensive unreported preliminary experiments served as the basis for the choice of the fabrication procedures used.

Method of Fabrication

Milling. - The calculated molecular percentage of boron carbide (B_4C) and the chemical analysis reported by the supplier are:

	Calculated (percent)	Chemical analysis (percent)
Boron	78.28	78.15
Carbon	21.72	21.60
Iron	-----	.12

The B_4C powder in the form of 400 mesh plus fines was milled with hardened chrome-manganese steel balls in an $8\frac{1}{2}$ -inch-diameter mill operated at 40 rpm for a period of 120 hours. The mill was half filled with hardened steel balls and sufficient boron carbide powder added to fill the interstices between them. Napthalene was added to fill the mill to the nine-tenths level.

Previous experience at the Lewis laboratory has shown that, under these conditions, between 35- and 40-percent iron would be picked up by the powder during 120 hours of milling. The addition of iron to the bodies investigated was accomplished in this manner. The chemical analysis by weight percentage of the boron carbide-iron ceramal evaluated in this investigation is:

B	Fe	C	Mn
48.90	36.40	13.75	0.30

Upon completion of the milling period, the milled material was filtered to remove excess liquid and was air-dried for 2 days. The filtering and drying operations tended to lump the particles, so the powder was then screened through a 200-mesh screen to crumble it for compacting.

Compacting. - The processed powder was poured into containers made of surgical rubber tubing $3/4$ inch in diameter. Rubber stoppers cemented in the ends were used as seals. The cylinder used for the hydrostatic pressing operation was made of 6140 steel hardened and drawn to Rockwell C-52 hardness. The specimen chamber was $1\frac{1}{2}$ inches in diameter and 8 inches long. The outside dimensions of the cylinder were diameter, 5 inches, and length, 10 inches. A 120,000-pound hydraulic press was used to apply pressure to the chamber. The specimens were compacted at a pressure of 10,000 pounds per square inch.

Sintering. - The furnace used for sintering (fig. 1) had a graphite resistance tube with a 3-inch inside diameter and a 5-inch outside diameter as the heating element. The atmosphere of the furnace was dried oxygen-free helium flowing at the rate of 40 cubic feet per hour, which produced a positive pressure of approximately 2 inches of water in the furnace shell. Furnace temperature was controlled with an automatic-recording total-radiation optical pyrometer. The sighting lens of this instrument was located on the top center section of the furnace shell. The instrument measured the temperature of the bottom end of the graphite sighting tube, which is located above the resistor tube. Sensitivity of the control instrument was such that specimen temperature, measured with an optical pyrometer by sighting through a quartz window mounted in the door of the loading chamber, varied a maximum of $\pm 20^\circ$ F. Compacts were heated to 1000° F in approximately 2 hours, brought up to the sintering temperature of 3750° F in an additional 4 hours, and held at that temperature for 1 hour. On completion of the heating portion of the sintering cycle, power to the furnace was cut off and the specimens allowed to furnace-cool for 16 to 20 hours to approximately 400° F; they were then removed from the furnace and air-cooled to room temperature.

Methods of Evaluation

Modulus of rupture. - The specimens used for modulus-of-rupture evaluation were formed by grinding the cylindrical bodies longitudinally, using diamond-embedded grinding wheels. The finished bars were approximately $1/4$ inch thick, $1/2$ inch wide, and $3\frac{1}{2}$ inches long. The three-point loading apparatus used with a 1-inch span for room-temperature modulus-of-rupture determination is shown in figure 2.

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The elevated-temperature short-time modulus-of-rupture apparatus (fig. 3) consisted of a commercial globar resistor furnace into which a lever-loading system had been incorporated. The specimen was supported on silicon-carbide knife edges 2 or $2\frac{1}{2}$ inches apart cemented to a refractory brick. All knife edges were rounded to a $1/8$ -inch radius to minimize stress concentrations at the contact points. Loading was accomplished by running water into a container hung from a lever arm supported by the upper knife edge, which was located midway between the supporting knife edges. The specimens were loaded at rates between 750 and 1500 pounds per square inch per minute.

As the specimen fractured, the loading arm fell and actuated a solenoid valve, which shut off the flow of water. Inaccuracy of weighing coupled with error caused by the excess of water that flowed while the solenoid valve was closing introduced a maximum inaccuracy of 1.2 percent in the measurement of load.

Evaluations were conducted at 1600°, 2000°, 2400°, and 2600° F. Furnace temperature was controlled to $\pm 10^\circ$ F by a platinum - platinum 13-percent rhodium thermocouple located directly behind the specimen and connected to a self-balancing recording potentiometer, which regulated power input to the furnace.

Specimen temperature was measured by a chromel-alumel thermocouple (positioned on the center of the specimen) at 1600°, 2000°, and 2400° F and with an optical pyrometer sighted on the center of the specimen at 2600° F. The difference between indicated furnace temperature and specimen temperature was of the order of 20° F.

After the specimen was placed on the supporting knife edges in the furnace, not more than 5 minutes was required for the specimen to heat to the evaluation temperature. A period of 10 minutes was allowed before loading was started, however, to insure that the specimen had reached equilibrium temperature conditions. All modulus-of-rupture evaluations were made in an oxidizing atmosphere.

Modulus-of-rupture strength for both room-temperature and elevated-temperature evaluations was calculated from the equation

$$S = \frac{3}{2} \left(\frac{pd}{wt^2} \right)$$

where

- S modulus-of-rupture strength, pounds per square inch
 p load on specimen (measured load times lever ratio), pounds
 d distance between supporting knife edges, inches
 w specimen width, inches
 t specimen thickness, inches

Thermal shock. - For evaluation of resistance to fracture by thermal shock, specimens broken in the room-temperature modulus-of-rupture runs were used. These pieces were made into specimens approximately 1/2 inch square by 1/4 inch thick. In this evaluation, the specimen was heated in a preheated globar electric furnace (held at the evaluation temperature) for 5 minutes, removed from the furnace with preheated tongs at the end of the heating period, and quenched in an air stream at a temperature of 72° F for 1 minute. The air stream was issued from an airline through a nozzle of 1/8 inch diameter, at a pressure of 100 pounds per square inch. The nozzle was located 2 inches from and directed toward the center of the specimen. The specimen was visually examined after each shock cycle. Presence of a crack in the specimen constituted a failure. Specimens were subjected to the cyclic treatment until failure occurred or until 25 cycles were completed at 1800° ± 10° F, followed by successive evaluation at 2000° ± 10° F.

Density measurements. - The measured density of each ceramal specimen evaluated was determined by use of the following equation:

$$\rho_s = \frac{W_s \rho}{(W_s - W) + W_w}$$

where

- ρ_s measured density of specimen, grams per milliliter
 W_s weight of specimen in air, grams
 ρ density of water at temperature of determination, grams per milliliter

W weight of specimen in water including weight of wire, grams

W_w weight of wire in air used to suspend specimen when weighed in water, grams

All weighings used for this determination were performed on an analytical balance. The individual values are believed to be correct to within ± 0.01 gram per milliliter.

Densities of the specimens were calculated by assuming that a mechanical mixture of the ingredients existed. Constituents other than iron and manganese were assumed to be boron carbide. The equation used for the calculation is

$$\rho_l = \frac{100}{\frac{a_c}{\rho_c} + \frac{a_i}{\rho_i} + \frac{a_m}{\rho_m}}$$

where

ρ_l calculated density of ceramal body, grams per milliliter

a_c weight percentage of boron carbide determined by difference

ρ_c density of boron carbide, grams per milliliter

a_i weight percentage of iron as determined by chemical analysis

ρ_i density of iron, grams per milliliter

a_m weight percentage of manganese as determined by chemical analysis

ρ_m density of manganese, grams per milliliter

Microscopic examination. - Representative specimens were selected for study of the structure of the ceramal. The specimens were sectioned using diamond-embedded cut-off wheels, and the surface was prepared for examination using standard metallographic wheels and diamond abrasives. Standard metallographic methods with reflected light were used for the microstructure studies.

Oxidation measurements. - Pieces from the modulus-of-rupture bars evaluated at room temperature were used for oxidation determinations. These specimens were measured with a micrometer and then

so placed on a refractory brick with V-shaped notches cut into the surface that the specimens touched the brick only at two lines. The brick and specimens were held at $1600^{\circ} \pm 10^{\circ}$ F in a globar furnace for 24 hours. After this exposure the specimens were removed, measured, and weighed. The specimens were again placed in the furnace for a period of 72 hours. At the end of this period, the specimens were removed and weighed. The loss in weight per unit time over the 72-hour period was calculated. After the 1600° F treatment, the specimens were further oxidized at $2000^{\circ} \pm 10^{\circ}$ F for 24 hours. At the end of this period the specimens were removed and weighed. The loss in weight per unit time over this 24-hour period was calculated.

RESULTS AND DISCUSSION

Fabrication

The procedure used in the fabrication of the boron carbide-iron ceramals was arrived at by evaluating the physical properties of specimens after various fabrication procedures were tried and by studying the microstructure of representative specimens.

Reference 2 shows that bonding takes place between boron carbide and iron at 3000° F and it was initially expected that sintering would take place at this temperature. Although the reaction between boron carbide and iron takes place at 3000° F, a higher temperature was required for the fabrication of a sound body in order to form a continuous carbide network with the portion of the boron carbide that does not take part in the reaction. The bodies appeared soft and powdery when sintered for short times at temperatures as high as 3500° F. Specimens exhibiting the best strength properties were obtained by sintering at 3750° F for 1 hour. Figure 4 shows the microstructure of specimen 29BC7, which was sintered at 3750° F for 1 hour. Specimens sintered at higher temperatures, for which no data are reported, showed carbide grain growth and were weaker than specimens sintered at 3750° F.

Evaluation

Modulus of rupture. - The results of the modulus-of-rupture evaluations of the ceramals and of the ceramic are presented in tables I and II, respectively. The trends in modulus-of-rupture

strength with temperature for the two materials are shown in figure 5. Peak strength values are used in this comparison, because it is assumed that these values more nearly represent the maximum potentialities of the experimental materials.

Strength of the ceramal declines slowly as temperature increases from room temperature to 2400° F. The loss in strength is approximately one-fourth over this range. At 2600° F the material has lost less than half its room-temperature strength, even though iron is very weak at this temperature. The high percentage of strength retention can probably be attributed to: (a) strength of carbide network, and (b) strengthening of the iron by solution with some of the boron carbide.

Strength of the boron carbide also declines slowly in the temperature range from room temperature to 2600° F. It can be seen from figure 5 that at all temperatures the strength of the ceramic exceeds that of the ceramal. Possible reasons for the lower strength of the ceramal are: (a) tendency of the metal addition to prevent the formation of a homogeneous carbide body, instead a carbide network, which undoubtedly is weaker is formed; and (b) presence of the weaker (metal-rich) phase.

Reference 2 shows that a bonding phase is formed between iron and boron carbide. The high percentage of strength retained at 2600° F indicates that the bonding phase has a higher softening temperature than the iron added to the ceramal, but figure 5 indicates that it is still weaker than the ceramic. These data verify the hypothesis of reference 2.

Thermal shock. - The thermal-shock evaluation indicated that the resistance to fracture by thermal shock of hot-pressed boron carbide is improved by the addition of iron. Two boron carbide specimens withstood 3 and 9 cycles at 1800° F, whereas two ceramal specimens withstood 25 cycles at 1800° F and 7 and 15 cycles at 2000° F. These results indicate that, as in the case of the titanium carbide ceramal (reference 5), the approximately 15 percent by volume addition of metal yielded a body that was superior in resistance to fracture by thermal shock to the pure ceramic. This improvement may have been achieved by the alteration of such physical properties as ductility and thermal conductivity (reference 6).

Density. - Measured densities of the ceramal specimens are listed in table I. The maximum variation in density of the ceramals

investigated was less than 5 percent. The calculated density, based on the assumption that the constituents are in the form of a mixture, is 3.36 grams per milliliter, which is 3.7 percent higher than the average measured density of 3.24 grams per milliliter. This difference in density can be attributed to: (a) formation of a new phase that is lower in density, making the density of the sintered body lower than that calculated for a mechanical mixture; or (b) porosity in the specimen causing lower density specimens even though the new phase has a density equal to or higher than that of the original mixture.

The microstructure of the ceramal (fig. 4), indicates the presence of at least two phases. The gray area is the boron-carbide matrix, the white area is the metallic phase, and the black spots are believed to be either pores or voids created during polishing. As previously stated, the iron was approximately 15 percent of the body by volume. This photograph, which depicts an area representative of the ceramals evaluated, shows more than 15 percent of the bonding material present. This disparity can be accounted for by assuming solution of the boron carbide in the iron, a chemical action between the two base materials, or a combination of both solution and chemical action.

The measured values of density of hot-pressed boron carbide (table II) are very close to that of a single crystal of boron carbide, which is 2.52 grams per milliliter (reference 7).

The strength-density ratio of boron carbide is compared with those of several titanium carbide-base ceramals in table III. The values presented show that the boron carbide ceramal is superior to the best titanium carbide ceramal by a factor greater than two at 2400° F. The strength-density ratio is of importance when the effects of stresses induced in rotating parts are considered.

Oxidation. - A comparison of the resistance to oxidation of boron carbide and the boron carbide-iron ceramal is shown in table IV. Both of these materials, in oxidizing, form a glassy surface coating of boron oxide that sublimates at the temperatures of interest. The preoxidizing treatment (24 hr at 1600° F) was used to permit the formation of the glassy phase and allow the surfaces to reach equilibrium. After this treatment no change in dimensions could be measured. The rates of weight loss presented therefore represent the combined effects of oxide formation and sublimation at equilibrium conditions. The coating formed was very thin and during long exposure did not become thicker.

At 1600° F the unit weight loss of the ceramic was about one and one-half times greater than that of the ceramal; however, at 2000° F the unit weight loss of the ceramal was about six times that of the ceramic. This inferiority of the ceramal at 2000° F is probably caused by reaction of the boron oxide with the iron, which produced a glassy phase that sublimed at a higher rate. Neither of the materials is suitable for use over long periods of time at temperatures above 1600° F in an oxidizing atmosphere without a protective coating.

CONCLUDING REMARKS

The boron carbide-iron ceramal evaluated in this investigation had good strength properties at temperatures up to 2600° F. This finding indicates that ceramals containing materials which form a bonding phase having a higher softening temperature than that of the metal addition may possess desirable strength properties at temperatures approaching the melting point of the metal. A comparison of the oxidation resistances of the two materials evaluated showed that the resistance of a ceramal may be different from that of the ceramic constituent. If a ceramal has undesirable oxidation characteristics, however, it may be usable providing a suitable coating is applied.

Inasmuch as ceramals apparently have characteristics that represent a compromise between the high strength and poor thermal-shock resistance of the ceramic constituent and the poor elevated-temperature strength and good thermal-shock resistance of the metal constituent, preliminary evaluation of a ceramal for a specific application should consider the relative importance of the two characteristics.

SUMMARY OF RESULTS

An investigation of boron carbide and of a ceramal comprising 36-percent (by weight) iron and 64-percent boron carbide yielded the following results:

Microscopic examination showed the ceramal to be composed of at least two phases: boron carbide, and a solution or chemical combination of iron and boron carbide. Ceramals containing materials that form a bonding phase may possess desirable strength properties at temperatures approaching the melting point of the metal.

The hot-pressed boron carbide was superior to the ceramal in modulus-of-rupture strength (50,300 to 39,100 lb/sq in. for the ceramal at room temperature, and 28,900 compared with 23,400 lb/sq in. for the ceramal at 2600° F). The high percentage of room-temperature strength retained by the ceramal at 2600° F can be attributed to the high strength of the carbide network, strengthening of the iron by combination with some of the boron carbide, or both. The ceramal was superior to the boron carbide in resistance to thermal shock. At 1600° F, the resistance to oxidation of the ceramal was better than that of the ceramic; although both materials oxidized rapidly at 2000° F, the ceramic was superior. The poor oxidation resistance of the ceramal indicated that a protective coating would be required for continued operation above 1600° F.

Density of the ceramal varied from 3.17 to 3.29 grams per milliliter. The low density of the ceramal gives it an additional advantage when it is compared with metals or other ceramals on the basis of strength-density ratio.

Lewis Flight Propulsion Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, November 17, 1949.

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TABLE I - MODULUS OF RUPTURE OF BORON CARBIDE-IRON
CERAMAL AT ROOM AND ELEVATED TEMPERATURES

Speciman	Span (in.)	Measured density (gm/ml)	Evaluation temperature (°F)	Modulus of rupture (lb/sq in.)
27BC6	1	-----	Room	38,100
29BC7	1	3.29	Room	39,100
29BC7	1	3.29	Room	30,900
29BC2	2.5	3.18	1600	31,000
29BC1	2.5	3.20	1600	32,600
29BC6	2.0	3.27	2000	32,000
29BC10	2.0	3.25	2000	27,400
29BC4	2.0	3.26	2400	28,600
29BC5	2.0	3.27	2400	25,100
29BC3	2.0	3.17	2600	23,400

TABLE II - MODULUS OF RUPTURE OF HOT-PRESSED BORON
CARBIDE AT ROOM AND ELEVATED TEMPERATURES

Speciman	Span (in.)	Measured density (gm/ml)	Evaluation temperature (°F)	Modulus of rupture (lb/sq in.)
9BC	1	2.51	Room	50,300
9BC	1	2.51	Room	46,900
5BC	2.5	2.50	1600	35,200
4BC	2.5	2.49	1600	35,300
6BC	2.5	2.50	2000	34,600
2BC	2.5	2.50	2000	29,800
7BC	2.5	2.50	2400	27,900
3BC	2.5	2.49	2400	36,000
8BC	2.5	2.51	2600	28,900

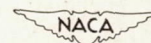


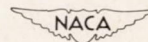
TABLE III - STRENGTH-DENSITY RATIOS OF BORON CARBIDE- AND
TITANIUM CARBIDE-BASE CERAMALS

Material (nominal composition by weight)	Measured density (gm/ml)	Modulus-of- rupture strength at 2400° F (lb/sq in.)	Strength- density ratio at 2400° F
64-percent boron carbide 36-percent iron	3.26	28,600	8773
90-percent titanium carbide 10-percent molybdenum	^a 5.12	^a 20,900	4082
90-percent titanium carbide 10-percent tungsten	^a 5.05	^a 19,000	3762
95-percent titanium carbide 5-percent cobalt	^a 5.09	^a 2,900	570
80-percent titanium carbide 20-percent cobalt	^a 5.35	^a 2,400	449

^aData from reference 4, tables IV, V, and VI.

TABLE IV - COMPARISON OF RESISTANCE TO OXIDATION OF
HOT-PRESSED BORON CARBIDE AND BORON-CARBIDE
CERAMAL CONTAINING IRON

Composition	Unit weight loss ((gm/sq cm)/hr)	
	1600° F	2000° F
Boron carbide	0.0000853	0.00154
Boron carbide con- taining iron	.0000579	.00912



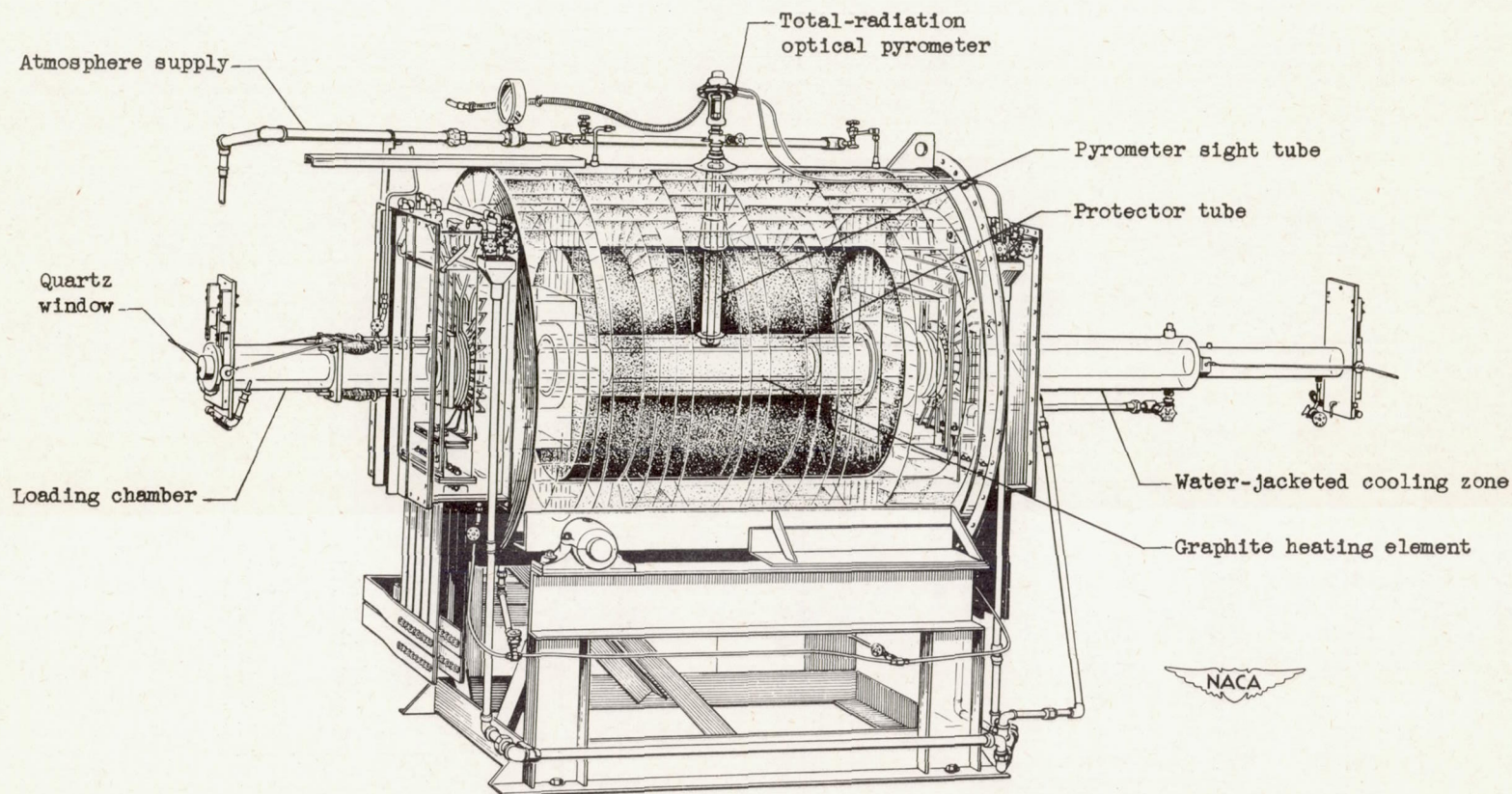


Figure 1. - Graphite-resistance electric furnace used for bonding study.

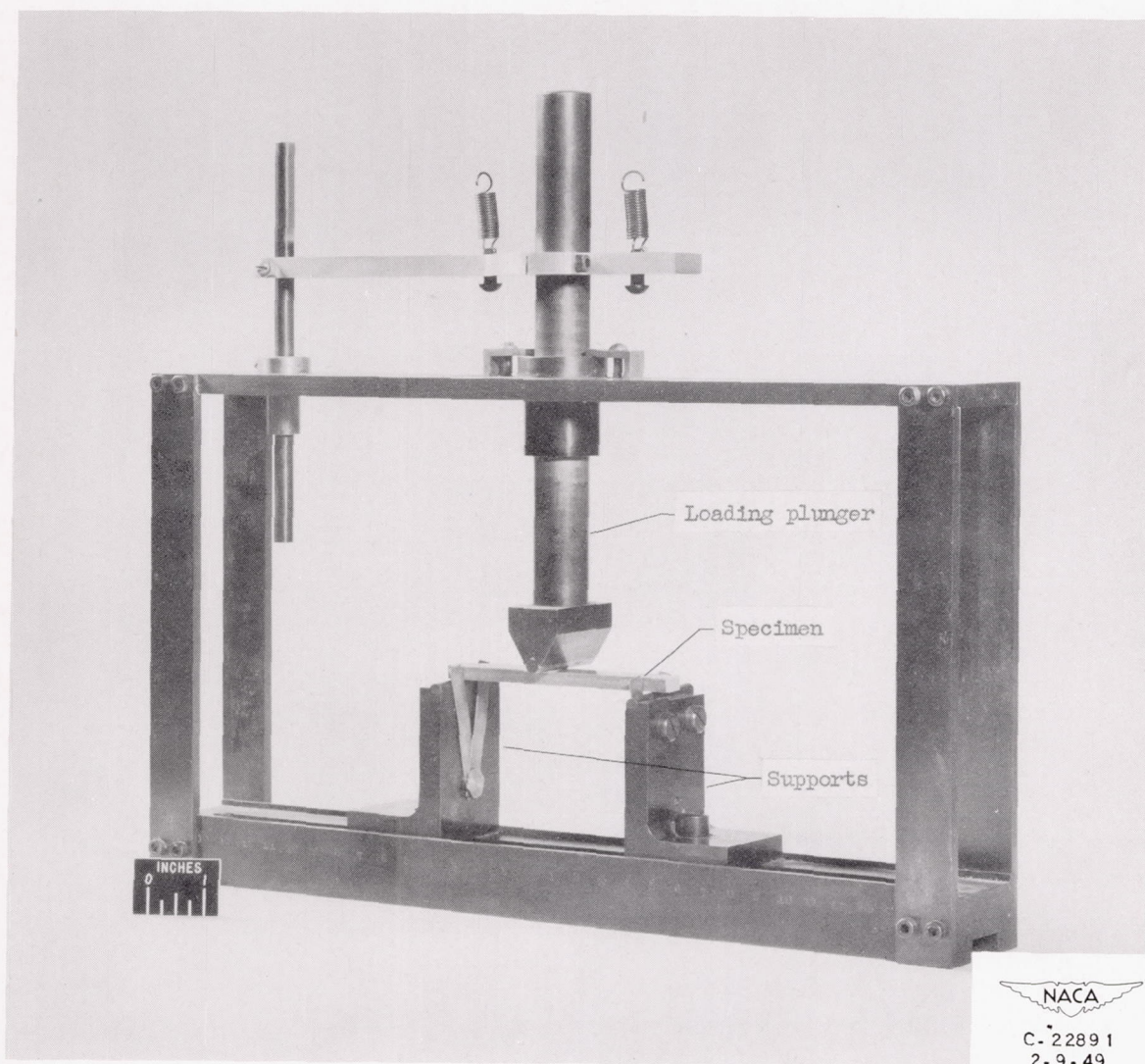


Figure 2. - Apparatus used for evaluation of modulus of rupture at room temperature.



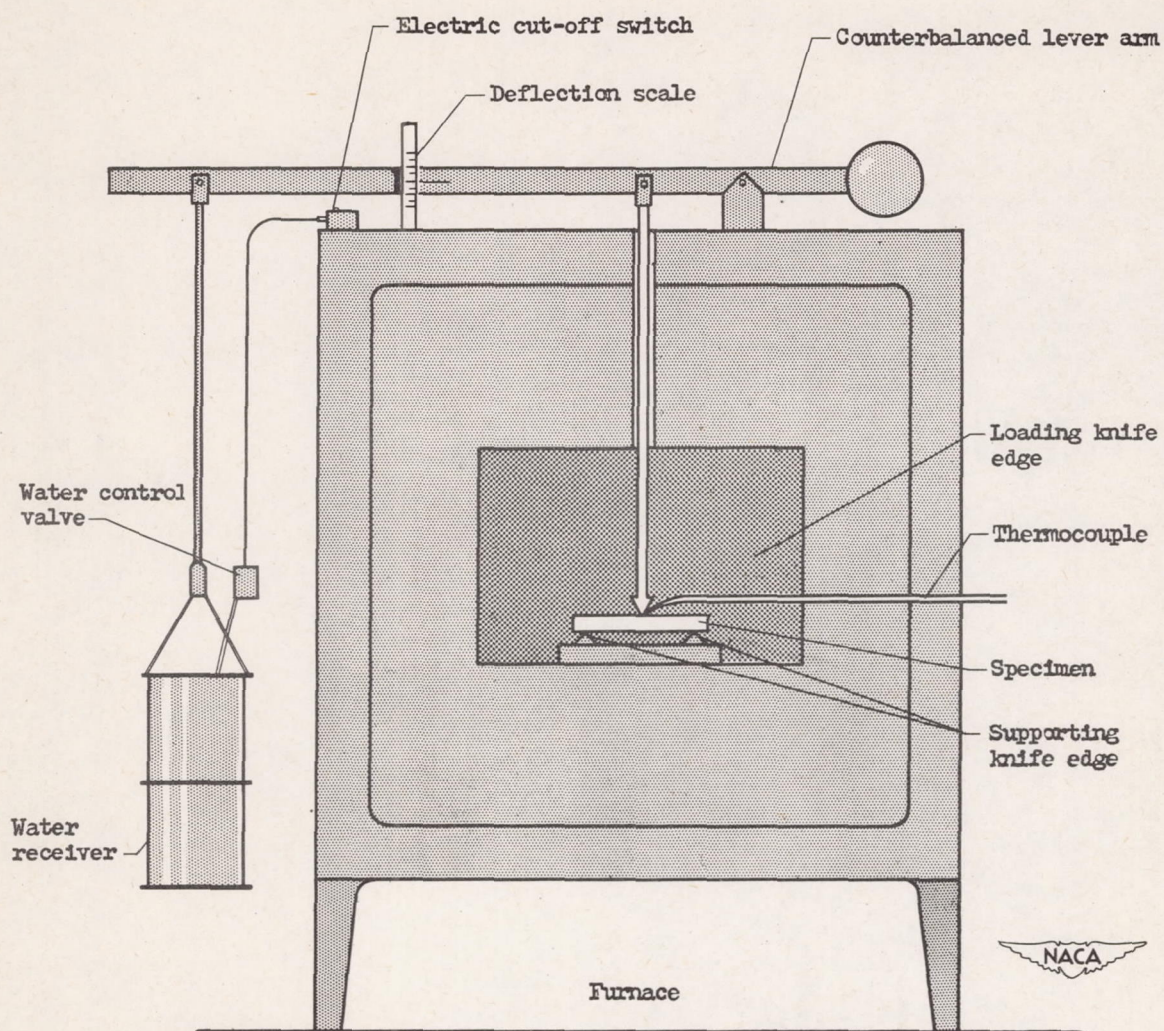
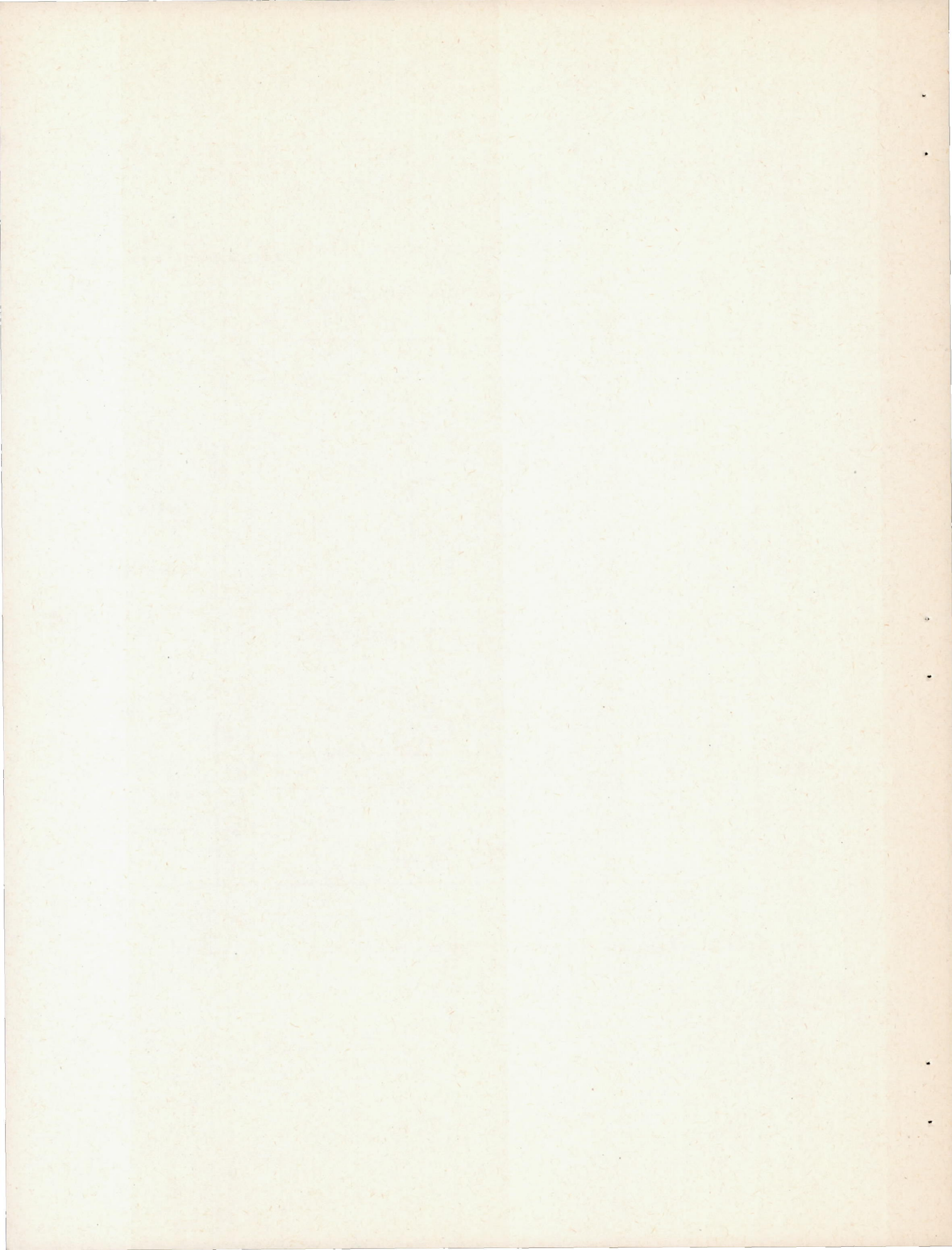
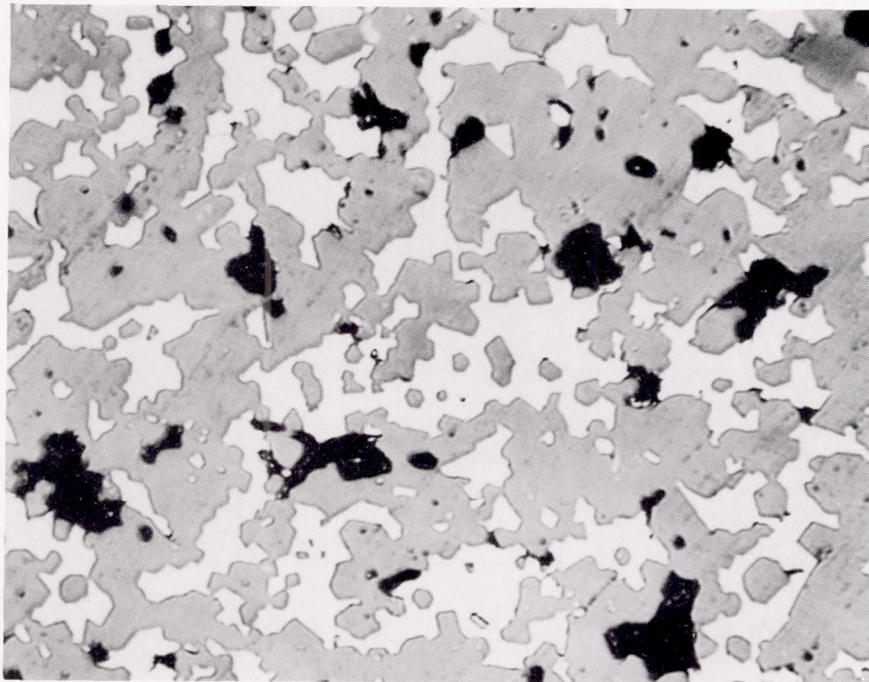


Figure 3. - Apparatus for modulus-of-rupture determination at high temperatures.





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Figure 4. - Photomicrograph of boron carbide-iron ceramal 29BC7 sintered at 3750° F for 1 hour. Gray area, boron carbide; white area, bonding phase; unetched. X1000.



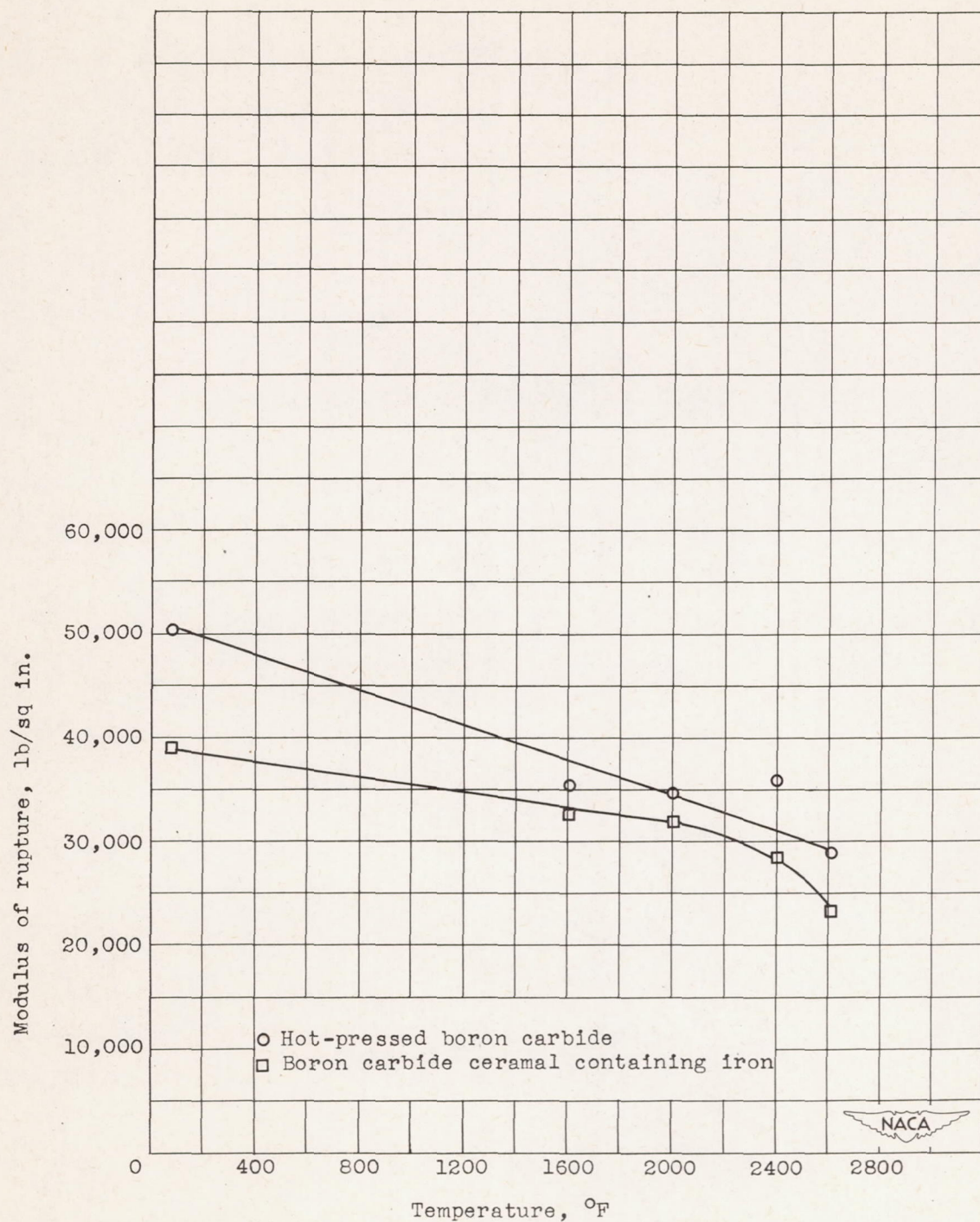


Figure 5. - Variation of modulus of rupture with temperature.

